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Form 1/77

Patents Act 1977

1 Title of invention

- 1 Please give the title of the invention **FLOOR COVERINGS**

2 Applicant's details

- ☐ First or only applicant

- 2a If you are applying as a corporate body please give:

Corporate name
Foxbo-Nairn Limited

Country (and State of incorporation, if appropriate) **England UK**

- 2b If you are applying as an individual or one of a partnership please give in full:

Surname

Forenames

- 2c In all cases, please give the following details:

Address **Horsley House
The Regent Centre
Gosforth
Newcastle Upon Tyne**

UK postcode (if applicable) **NE3 3PZ**

Country **United Kingdom**

ADP number (if known)

6613046001 P.A.

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☐ Second applicant (if any)

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↓
please give details below

Agent's name CRUIKSHANK & FAIRWEATHER

Agent's address 19 Royal Exchange Square
Glasgow

Postcode G1 3AE

Agent's ADP
number 547002

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① Reference number

4 Agent's or
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❶ Claiming an earlier application date

5 Are you claiming that this application be treated as having been filed on the date of filing of an earlier application?

Please mark correct box

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☐ filing date

day	month	year
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15(4) (Divisional) ☐ 8(3) ☐ 12(6) ☐ 37(4) ☐

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Country of filing	Priority application number (if known)	Filing date (day, month, year)
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- 7 The answer must be 'No' if:
- any applicant is not an inventor
 - there is an inventor who is not an applicant, or
 - any applicant is a corporate body.

Please supply duplicates of claim(s), abstract, description and drawing(s).

Please mark correct box(es)

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8a Please fill in the number of sheets for each of the following types of document contained in this application.

Continuation sheets for this Patents Form 1/77

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Claim(s)

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Description

16

Abstract

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Drawing(s)

2

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Priority documents (please state how many)

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Translation(s) of Priority documents (please state how many)

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Patents Form 7/77 - Statement of Inventorship and Right to Grant (please state how many)

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Patents Form 9/77 - Preliminary Examination/Search

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Patents Form 10/77 - Request for Substantive Examination

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Request

We request the grant of a patent on the basis of this application.

Andrew & Farweather
Signed

Date 04.08.1994

day month year

(Agents)

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FLOOR COVERINGS

The present invention relates to floor coverings and more particularly to tile or sheet form floor coverings made of one or more layers of polymers.

Most floor coverings of this type are based on polyvinylchloride (PVC) polymer. In more detail, PVC polymer resin is generally mixed with a plasticiser (solid or liquid) (usually with various other additives such as fillers, polymer stabilisers, and processing aids) to form a spreadable paste which can be formed into sheets by spread coating using knife or roller coater equipment and then thermally cured e.g. by oven heating.

The use of PVC does however raise significant environmental problems due to the use of chlorine and there is accordingly a need for floor coverings based on alternative polymers. Polyalkene polymers are generally preferred from an environmental point of view but the use of conventional polyalkenes presents significant processing problems and they are not suitable for use in floor covering manufacturing facilities based on spread coating and calendering technology.

It is an object of the present invention to avoid or minimize one or more of the above disadvantages.

It has now been found that a particular class of polyalkenes can be successfully used in floor covering manufacture based on more or less conventional spread coating or calendering technology. More particularly, suitable polyalkenes in accordance with the present invention are those having a relatively narrow molecular weight distribution (MWD) and, a small amount of long chain branching and produced by single site (constrained geometry) catalysed polymerisation, and having the following characteristics:

- a) Melt Index (MI) of from 0.1 to 100
- b) Density of from 0.86 to 0.97; and
- c) a DRI of from 0.1 to 6.0, preferably from 0.4 to 5.5.

As used herein the following terms have the meanings indicated:

Melt Index (MI) or I_2 is the amount (in grams) of polymer resin which is extruded in a predetermined period of time (10 minutes) as measured in accordance with ASTM (American Standard Testing Method) D-1238 (190/2.16).

Molecular Weight Distribution (MWD) is the ratio of weight average molecular weight (Mw) to number average molecular weight (Mn) (i.e. Mw/Mn).

Density is the mass (in grams) of 1 cubic centimetre of resin as measured in accordance with ASTM D-792 standard.

Dow Rheology Index (DRI) is an index of long chain branching measured by comparing the shift to the right (due to a longer relaxation time), relative to a polymer resin with zero long-chain branching (LCB), in a plot of zero shear viscosity against relaxation time (both from a cross viscosity equation).

Other abbreviations used herein which are common in the art include:

PHR - parts per hundred parts by weight of polymer resin (or principal polymer resin component).

Thus in one aspect the present invention provides a sheet material suitable for use in or as a floor covering and comprising a polyalkene resin in intimate admixture with one or more additives selected from a filler and a spread coating processing aid, wherein said polyalkene resin has a relatively narrow molecular weight distribution (MWD) and, a small amount of long chain branching and produced by single site (constrained geometry) catalysed polymerisation, and having the following characteristics:-

- a) Melt Index (MI) of from 0.1 to 100
- b) Density of from 0.86 to 0.97; and
- c) a DRI of from 0.1 to 6, preferably 0.4 to 5.5.

Whilst processing aids may be used in the new materials of the present invention, to adjust or accentuate particular processing characteristics, it is a feature of the polyalkene resins used that they do not require the use of a plasticiser thereby significantly reducing environmental problems associated with the use of plasticisers.

Various polyalkene resins suitable for use in the materials of the present invention are known in the art. In general they are produced by polymerisation of alkene monomers in the presence of particular catalysts which restrict the progress of the polymerisation and are known as metallocenes (the resulting polymers being commonly referred to as metallocene polyolefines conveniently abbreviated as MPOs). Such polyolefines and processes for their production are described in, inter alia, US Patent 5,272,236.

Suitable polyalkene resins that are commercially available from the Exxon Chemical company of USA and the Dow Chemical company of Midland, Michigan, USA, are listed in Tables 1 and 2 below.

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Table 1Exxon's EXACT (TM) ResinsProductKey Properties

EXACT 4006

Density 0.880

MI 10.0

EXACT 4003

Density 0.895

MI 9.0

EXACT 4023

Density 0.882

MI 35.0

EXACT 4033

Density 0.880

MI 0.80

Table 2 - Dow's INSITE (TM) TECHNOLOGY POLYMER (ITP)

<u>Product</u>	<u>Key Properties</u>	
Engage CL8200	Density	0.870
	MI	5.0
	DRI	0.5
Engage CL8150	Density	0.868
	MI	0.5
	DRI	2.0
Affinity SM1300	Density	0.902
	MI	30.0
	DRI	0.4
Affinity SM1250	Density	0.885
	MI	30.0

In a further aspect the present invention provides a polymer resin-based floor covering comprising at least one layer of a sheet material of the invention. It will be appreciated that in general such floor coverings comprise two or more different layers having particular functions, bonded together. Typically there may be included layers such as a foamed layer to provide cushioning; a structural layer comprising a reinforcing

carrier or substrate impregnated and/or coated with a saturant formula; a solid backcoat layer; and a clear protective or topcoat layer.

The sheet materials of the invention may be produced by a process comprising the steps of:

providing a suitable polyalkene resin in accordance with the present invention and one or more additives selected from a filler and a spread coating or calendering processing aid;

bringing said polyalkene resin into intimate admixture with said one or more additives in a high shear mixer and substantially free of plasticiser for a period of at least 10 minutes at an elevated temperature of at least 75°, preferably from 100 to 250°C, most preferably from 130 to 200°C, for melting the polyalkenes and sufficient to bring the mixture into a substantially fluid state without substantial degradation of the mixture; forming the fluid mixture into a sheet form; and allowing said sheet to cool and solidify. The fluid mixture may be formed into a sheet by any suitable technique including spread coating, calendering etc.

(
The sheet material production processes of the present invention have significant advantages over those made using conventional polyalkene or polyolefin resins. Apart from the superior processability which allows the

use of conventional existing production plant previously utilized for PVC resin based sheet materials with minimal modifications, they also have lower energy consumption costs due to the substantially reduced curing temperatures required as compared with PVC resin based production which involve increasing temperature to effect a thermal curing as opposed to a cooling to effect "crystallisation curing" by "solidification". Further benefits that can be obtained in relation to particular floor covering layers in products of the invention include better toughness of the outer clear coat layer with better impact resistance resulting from the lower crystallinity associated with lower density; better cell recovery in foamed cushioning layers; and better filler acceptance due to more homogenous nature of the polymer (narrow MWD); and good flowability of the saturated layer resulting from high MI with little or no comonomer blocking.

In relation to the various aspects of the present invention it will be appreciated that other polymer resins outside those specified may be used in admixture with the specified ones e.g. in order to "extend" the specified polyalkene resin for reasons of economy by using a cheaper polyalkene resin, or to modify finish or other characteristics. The amount of such other polymer resin that may be used will depend primarily on how they

affect the fluidity and spread coating characteristics of the materials of the invention.

Additives that may be used in the materials of the present invention and the amounts thereof, will depend on the function and desired properties of the sheet material and may also, to some extent, depend upon the particular polymer resins used. Principal additives generally well known in the art, that may be mentioned include the following:

1. Whiting filler is used to increase opacity. Generally there is employed less than 500 PHR, preferably from 20 to 120 PHR in saturant formula and foamable cushioning materials and up to 200 PHR in solid backing layers.
2. Stabilisers e.g. hindered phenol at from 0.05 to 0.30 PHR are usually desirable, optionally with co-stabilisers e.g. organosulphur compounds such as DSTDP at from 0.2 to 1.0 PHR.
3. Soap for improving flowability e.g. zinc or calcium stearate at from 0.2 to 1.0 PHR.

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4. Blowing agent, preferably low temperature variety, such as azodicarbonamide is used for foamable cushioning layers at from 2.0 to 4.5 PHR, together with a suitable foaming activator such as zinc oxide.

5. Titanium oxide for pigmentation at from 2 to 6 PHR in foamable cushioning layers.

6. Crosslinking agent for modifying the MPO resin component, e.g. an organic peroxide at from 0.1 to 5.0 PHR, for increasing toughness and/or stiffness of the sheet layer.

7. Flame retardent is generally used at less than 50 PHR e.g. antimony oxide at from 2 to 10 PHR or halogen-free zinc oxide and boron oxide at from 5 to 20 PHR.

8. Polymer resins other than the specified MPOs may be used as noted above as extenders or modifiers in amount of from 10 to 30 PHR. Examples that may be mentioned include LLDPE (Linear Low Density PolyEthylene), EVA (Ethylene Vinyl Acetate), Ionomers e.g. SURLYN (TM) available from the DuPont Company, and VLDPE (Very Low Density PolyEthylene).

9. Other additives that may be mentioned include dyes, inks, antioxidants etc. which are generally used in relatively small amounts at less than 50 PHR.

10. Carriers or substrates used with saturant formulations may have various forms e.g. woven or non-woven mesh or fabric, or tissue, of more or less thermally stable materials such as glass fibre.

The polyalkene or polyolefin resins used in accordance with the present invention may be of various different types including random bipolymers and terpolymers, and block copolymers, based on a variety of monomer units including lower alkene, preferably 1-alkene, having from 2 to 8 carbon atoms (e.g. propylene but most preferably ethylene; dienes; cycloalkenes; and vinyl aromatic compounds.

Further preferred features of the invention will appear from the following detailed Examples given by way of illustration and the accompanying schematic drawings in which:

Fig. 1 is a schematic side view showing a first part of a floor covering production line; and

Fig. 2 is a similar view of the second part of the production line of Fig. 1.

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Fig. 1 shows a first-stage production line 1 for producing a first-stage three layer sheet material 2 by applying saturant, foam gel, and back-coat layer formulations 3, 4, 5 onto a glass fibre tissue web (approx. 0.45 mm thick) 6 supplied from a supply drum 7 via a first accumulator 8. The tissue web is passed via a first weight/unit area measuring system 9 to a first spread coating unit 10 at which the hot melt saturant formulation 3 (at approx. 90°C) is applied onto one side 11 of a first roller 12 to a predetermined thickness of about 0.55 mm controlled by a first knife 13, from a first continuous high shear farrel-type mixer 14. At the other side 15 of the first roller 12, the saturant formulation is transferred to the tissue web 6 at a nip 16 between the first roller 12 and an opposed tissue web support roller 17. The impregnated tissue web 18 is then passed around a large diameter chilled drum 19 set for a surface temperature of around 25 to 40°C and further smaller diameter cooling drums 20 for "crystallisation curing" or solidification.

The hot melt foam gelling and back-coat layer formulations 4, 5 are then successively applied to the coated tissue web 18 at approximate thickness of 0.2 and 0.6 mm, respectively, in generally similar manner at second and third spread coating units 21, 22, except that a large diameter chilled drum 19 is omitted at the

back-coat layer stage. The resulting three layer sheet material 2 is then collected on a wind-up drum 23 down-stream of a second accumulator 24. If desired this sheet material is then passed to a rotogravure or other printing station for application of graphic design material etc. in generally known manner, for example, using ink designed for chemical embossing.

Fig. 2 shows a second stage production line 101 in which like parts corresponding to those in Fig. 1 are indicated by like reference numerals to which have been added 100. The three layer sheet material 2 produced in the first stage production line 1 is supplied from a supply drum 107 via an accumulator 108 to a fourth spread coating unit 110 at which a clear coat formulation 125 is applied to said sheet material 2 at a thickness of about 0.2 mm, and cured as before except that in this case a heat shield 126 is provided between the chilled drum 119 and the hot mixer 114 to help improve temperature control etc.

If desired a further foamed back-coat layer may be applied using yet another spread-coat applicator (not shown). It will incidentally be appreciated that in accordance with common practice in the industry the order of application of the various layers can be varied to a greater or lesser extent.

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Finally where a polish or lacquer type finish is required this can be applied using a grooved roller applicator 127.

The resulting multi-layer sheet material 140 is then passed through a multi-stage hot air oven 141 on a belt support 142 set to a maximum temperature of around 130°C with a dwell time of around 1½ minutes to allow foaming expansion of the foam gel layer (from about 0.2 mm to about 0.5 mm) with selective control thereof by chemical embossing where this is used, whereupon final cooling of the finished sheet material takes place at further cooling drums 120 prior to collection on the take-up drum.

Example 1 - Individual layer formulations

The following polymer resin formulations have been prepared:

<u>A. Clear coat layer</u>	<u>PHR</u>
MPO Resin Engage EP8500 (Dow Chemical Co.)	100
(MI 5.0, Density 0.87, DRI 0.5)	
Irganox 1010 Antioxidant Stabiliser	0.05
BHT Antioxidant Stabiliser	0.03
2,5 - TRI Cross-Linking Agent	0.1

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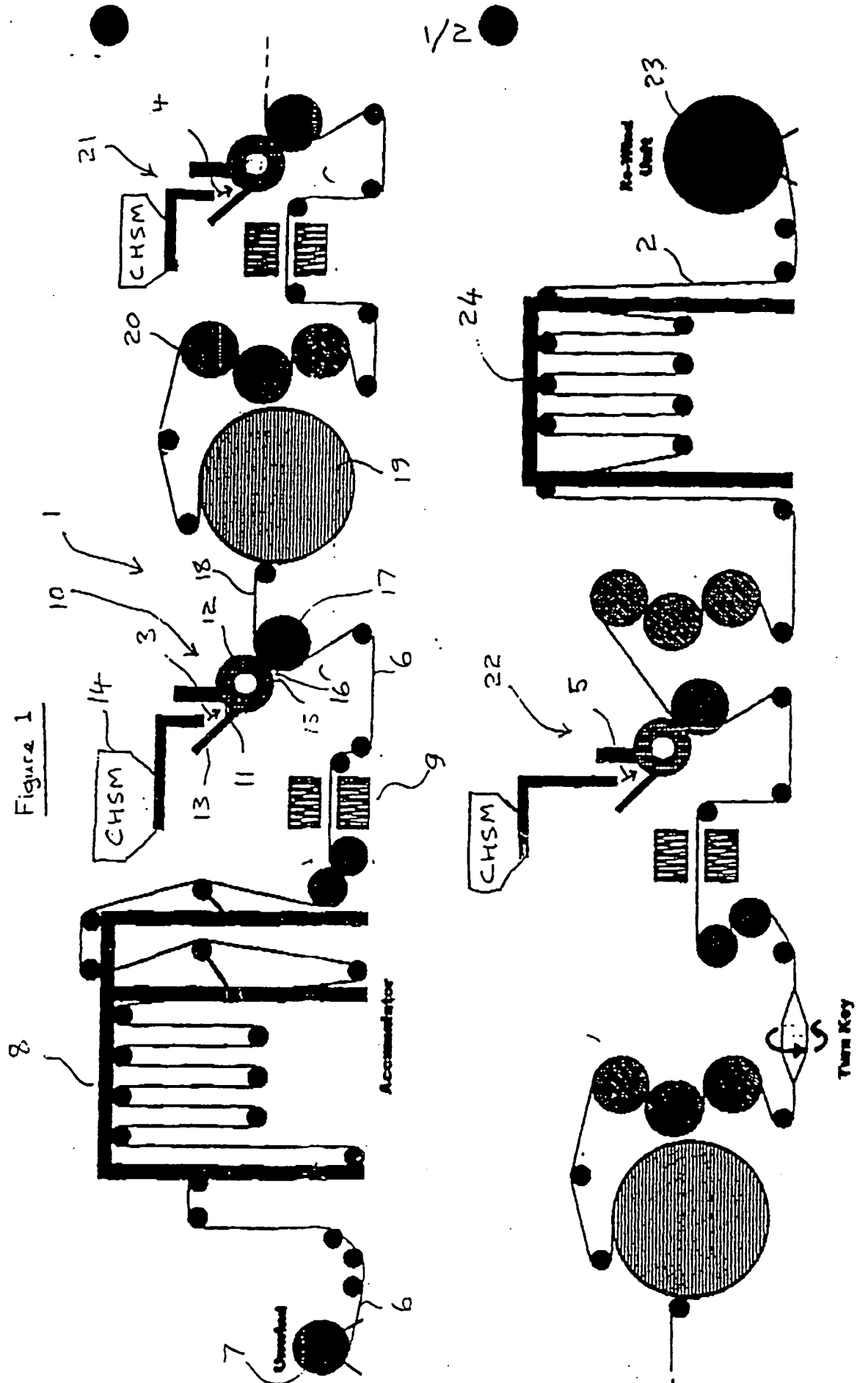
<u>B. Foamable Gel Layer</u>	<u>PHR</u>
MPO Resin Engage EP 8500 (Dow Chemical Co.)	100
Whiting filler (generic)	15
Azo blowing agent (generic)	3
Zinc oxide Foaming Catalyst	1.5
Titanium oxide pigmentation component	4
Irganox 1010 Stabiliser	0.075
DSTDP Stabiliser	0.05
Calcium stearate Flowing Agent	0.10
Firebrake (TM) flame retardant	5
Antimony oxide flame retardant	4
<u>C. Saturant Layer</u>	<u>PHR</u>
MPO Resin Engage EP 8500 (Dow Chemical Co.)	100
Whiting filler (generic)	50
Irganox 1010 Stabilizer	0.1
Zinc stearate Flowing Aid	0.4
<u>D. Solid Backcoat Layer</u>	<u>PHR</u>
MPO Resin Engage EP 8500 (Dow Chemical Co.)	100
Whiting filler (generic)	200
Titanium oxide pigmentation component	4
Irganox 1010 Stabiliser	0.075
DSTDP Stabiliser	0.05
Calcium stearate Flowing Agent	0.10
Firebrake (TM) flame retardant	5
Antimony oxide flame retardant	4

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Example 2 - Individual Layer Formulations

A further set of polymer resin formulations is prepared as in Example 1 above but with Dow Chemical Co.'s Affinity SM 1250 as the MPO Resin component in place of EP 8500.

Figure 1



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